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IMPROVED ELECTRICAL PROPERTIES OF EPOXY RESIN WITH NANOMETER-SIZED INORGANIC FILLERS (POSTPRINT)

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14. ABSTRACT
Nanometer-sized inorganic fillers are increasingly used as reinforcing materials for mechanical or thermal property improvement of polymers. Improvements in mechanical modulus or heat deflection temperature are often realized. These fillers may also improve some electrical properties such as corona endurance or dielectric breakdown voltage in polymers. In compact high voltage power supplies, epoxy resins are often the potting material of choice in manufacturing processes. This is often true for applications requiring a robust or position-insensitive insulation system design, such as mobile communications equipment or aerospace flight vehicles. Nanometer-sized inorganic fillers in epoxy resins can result in improved mechanical and electrical performance, without affecting the processes for component manufacturing. In our previous work, polyhedral oligomeric silsesquioxane (POSS) was selected as the nanometer-sized inorganic filler of interest. POSS-filled epoxies showed a five times improvement in ac corona lifetime for selected POSS-epoxy blends compared to unloaded epoxy.

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Improved Electrical Properties of Epoxy Resin with Nanometer-Sized Inorganic Fillers

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***Abstract*-Nanometer-sized inorganic fillers are increasingly used as reinforcing materials for mechanical or thermal property improvement of polymers. Improvements in mechanical modulus or heat deflection temperature are often realized. These fillers may also improve some electrical properties such as corona endurance or dielectric breakdown voltage in polymers.**

In compact high voltage power supplies, epoxy resins are often the potting material of choice in manufacturing processes. This is often true for applications requiring a robust or position-insensitive insulation system design, such as mobile communications equipment or aerospace flight vehicles. Nanometer-sized inorganic fillers in epoxy resins can result in improved mechanical and electrical performance, without affecting the processes for component manufacturing.

In our previous work, polyhedral oligomeric silsesquioxane (POSS) was selected as the nanometer-sized inorganic filler of interest. POSS-filled epoxies showed a five times improvement in ac corona lifetime for selected POSS-epoxy blends compared to unloaded epoxy [1]. In the current study, the average dielectric breakdown voltage of POSS-filled epoxy was increased thirty-four percent compared to unloaded epoxy. Additionally, scanning electron microscopy showed uniform dispersion of the POSS filler down to a level of 10-100 nm. Dispersion uniformity appears to be a critical parameter in obtaining the desired property enhancements.

I. INTRODUCTION

Nano-sized inorganic fillers have often resulted in improvements in specific properties for a variety of polymers. These nano-sized fillers may offer improved properties due to interfacial characteristics, size uniformity, or inhibition of agglomeration. Property enhancements may be electrical, mechanical, or thermal in nature. Besides increases in end use properties, with the small size of the additive, melt viscosity can remain similar to the base polymer allowing conventional processing [2].

The work of Nelson and Fothergill [3] showed significant improvements in electrical strength with relatively small percentages (10% by weight) of 23 mm titanium dioxide nanoparticles in epoxy. Cao, Irwin and Younsi [4] experimented with various blends of polyimide and alumina nanoparticles with filler percentages of 1% to 10%. These also exhibited an increase in breakdown strength over the base polymer. Henk et. al. [5], found an over nine times increased corona resistance with a 4.7% loading of nanometer sized silica particles in an epoxy when compared to the pure epoxy. In prior work with a thermoplastic [6], long-term breakdown studies were conducted using a control polypropylene and a 30% P0SS-loaded polypropylene. The thirty percent POSS loaded polypropylene showed a seven times increase in breakdown lifetime (with corona present) over the polypropylene without POSS.

In more recent work [1], long-term breakdown and corona resistance lifetime were investigated for a control epoxy and a 5% by weight POSS loaded epoxy. Additional measurements using infrared spectroscopy were done to garner information for possible performance explanations. Surface FTIR measurements were done on unexposed and corona exposed POSS-epoxy specimens, which confirmed that a silicon rich layer is developed on the POSS-epoxy sample surface, due to the corona exposure. In this paper, the short-term dielectric breakdown voltage of POSS-loaded epoxy samples was investigated. The primary focus was on samples with an additive of 5% POSS by weight, in an epoxy matrix.

II. EXPERIMENTAL METHOD

An epoxy resin from Resolution Performance Products was used (EPON 828). The curing agent was an aliphatic diamine (Jeffamine D400). All samples were cured for two hours at 80°C, then three hours at 125°C. Corona lifetime samples averaged 0.17 mm in thickness. Special effort was required to design a fixture for the manufacture of these relatively thin epoxy samples, while maintaining a consistent thickness without voids or holes. The structure of the particular POSS used, (trisilanolphenyl-POSS obtained from Hybrid Plastics), is shown in Figure 1.

The short-term dielectric withstand tests were performed with ASTM electrodes [7]. Each rod electrode was 6 mm in diameter with a 0.8 mm radius at the tip edge. The sample was placed between two identical, aligned rods. A special fixture was designed to hold the samples, inhibit surface flashover (without insulating gas or liquid immersion), and enable bulk breakdown. It is shown in Figures 2 and 3. Sixty Hz ac voltage was applied at a rate of approximately 1000 V/s, in a test circuit that limited the breakdown current to about 40 mA.

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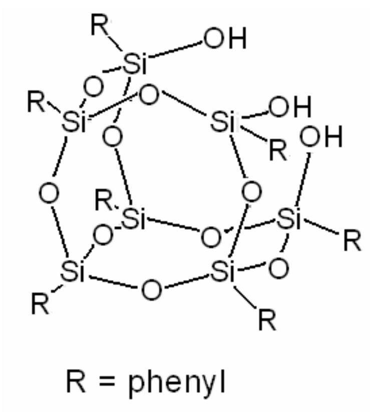

Figure 1: Trisilanolphenyl-POSS structure used in experiment.

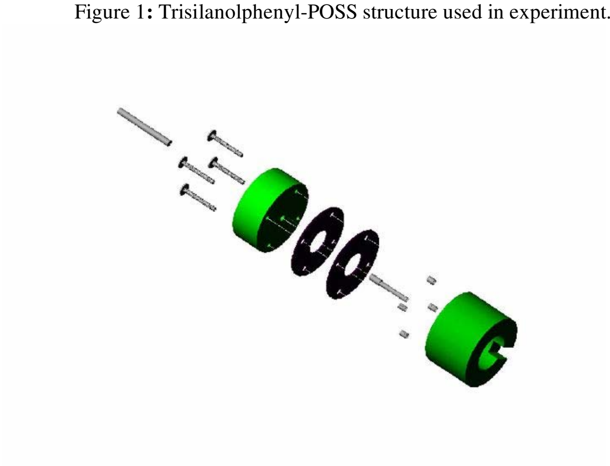

Figure 2: Custom sample holder: exploded view drawing

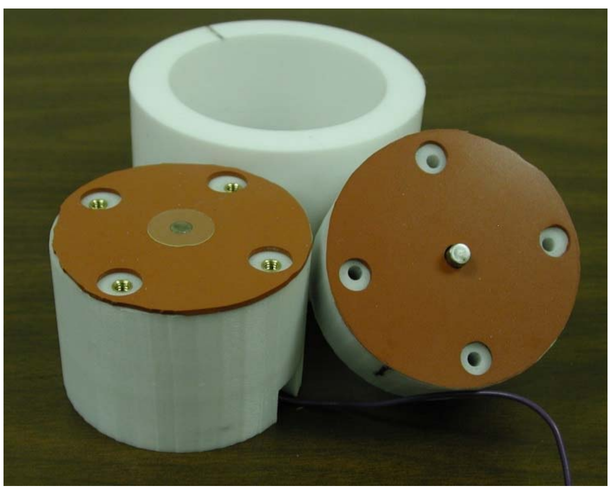

Figure 3: Custom sample holder: Photo of holder with 12.7 cm. dia. sample on lower electrode.

In order to ensure uniform dispersion of the nanomaterials throughout the epoxy matrix, it was necessary to employ a combination of thermal and mechanical agitation techniques during the blending process. To verify the dispersion uniformity, spot checks along fracture surfaces of randomly selected samples were performed by Scanning Electron Microscopy (SEM). High resolution SEM microscopy was done with a Hitachi S-4800 High Resolution Scanning Electron Microscope with an ultimate resolution of 1 nm.

III. Results and Discussion

Five percent POSS-loaded epoxy showed a thirty-four percent increase in dielectric breakdown voltage over the epoxy without POSS (Figure 4). Average breakdown voltage for the control epoxy was 17.7 kV rms. Average breakdown voltage for the POSS-epoxy was 23.7 kV rms.

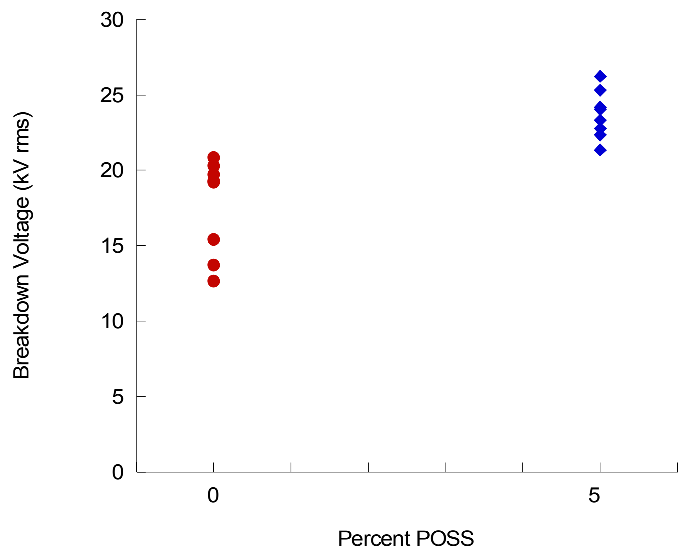

Figure 4. Dielectric breakdown voltage of control epoxy and 5% POSS loaded epoxy.

High resolution SEM studies show good dispersion of the nanofillers. The fracture surface in Figure 5 suggests that the POSS particles are in 10 to 20 nm clumps.

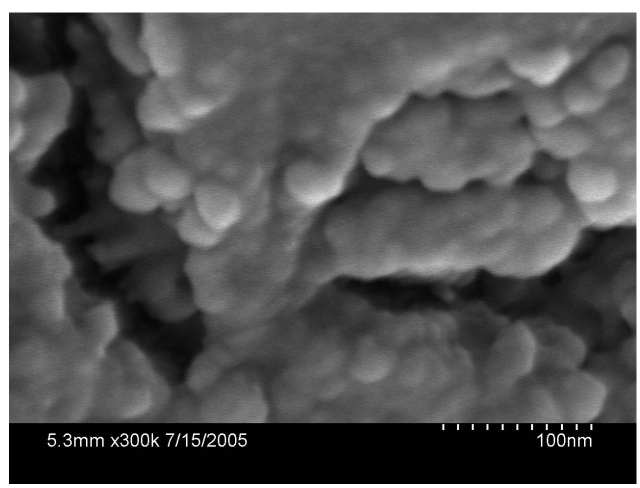

Figure 5. Fracture surface of 5% POSS-epoxy sample.

IV. Summary and Conclusions

POSS-epoxy samples showed improved dielectric breakdown voltage when compared to neat epoxy samples. The addition of inorganic nanoparticles to epoxies shows the potential of increasing voltage breakdown for the selected epoxy. The good dispersion, evidenced by SEM measurements on the samples tested, shows rationale for a correlation of the increase of dielectric withstand voltage with the use of small amounts of POSS nanoparticles in the neat epoxy. With reasoning taken from Nelson and Fothergill for their work on nanometer sized titanium dioxide in epoxy [3], POSS nanofiller in epoxy may allow localized charge movement thereby minimizing bulk charge accumulation. This minimizing of bulk charge accumulation may keep internal fields low and aid in increasing dielectric breakdown voltage.

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